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CHARACTERIZATION OF THE Sb_2O_3 THIN FILMS BY X -RAY SCATTERING

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Thin films of Sb_2O_3 have been prepared by thermal sputtering on a substrate of SiO_2. The X-ray scattering experiment from the Sb_2O_3 films was accomplished using a reflectometer. It consists of an X-ray source, a curbed multilayer monochromator on the incident beam side, a slit in front of the sample mounted on the center of the circle of the goniometer and two slits preceding the detector on a circle of the diffracted beam side. Specular X-ray scattering is sensitive normal to the sample surface. It provides vertical structural structure parameters (the density and roughness of the substrate and the density, thickness and roughness of the Sb_2O_3 layers on top of the SiO_2 substrate). The scanning of the surface was done at an incidence angle of the X-ray is in the range of 0-8° and the reflected intensity was recorded. The specular and diffuse X-ray scattering obtained from the deposed thin films, having different thickness, represent a method that is sensitive to density contrasts and can therefore be applied to all sorts of unpatterned surfaces and layered structures. It is applicable to structures on the nanometer scale and roughnesses on the subnanometer scale. The obtained data have shown that at lest ultrathin Sb_2O_3 layers reveals a gradated layer structure. The results can be used in controlling of the thin layers fabrication, thickness determination that have to rely upon densities and optical constant of layers.

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1. Introduction

The dielectric and semiconductor industry is increasing the wafer diameter for integrated circuit fabrication from 200 mm to 300 mm. According to Moore's law, the geometry of semiconductor devices is further reduced. The hystogram presented in Fig. 1 presents the progress of the "technology" in electronic industry [1, 2]. Frequently, the value of roughness is determined by atomic force microscopy. The value of roughness is important due to fact that in semiconductor technology, the gate length presents a decreasing tendency from 250nm to 180 nm. In consequence, the gate layer needs further perfection by reducing the interface and surface roughness.

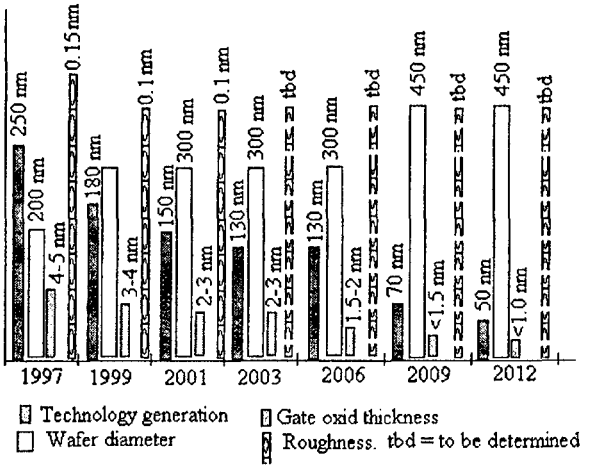

Fig. 1. The progress of technology and its consequences.

2. Sandwich structure

A sandwich structure is obtained by sputtering method and is typically in case of a transistor. The transistor performance is determined primarily by three parameters: gate length, gate dielectric

thickness and junction depth. Fig. 2 displays the schematic setup of a dielectric thin films deposited on a substrate having a typical microgeomety. For example, the thin gate oxides with oxide thickness < 10 nm, the Si /SiO_2 interface structure and the SiO_2 surface structure affect the insulating function of the SiO_2 layer [5]. More than, the channel mobility of electrical charge becomes smaller with increasing Si /SiO_2 interface roughness. Therefore, surfaces and interfaces have to be carefully monitored during wafer processing to keep them chemically clean and physically smooth [7].

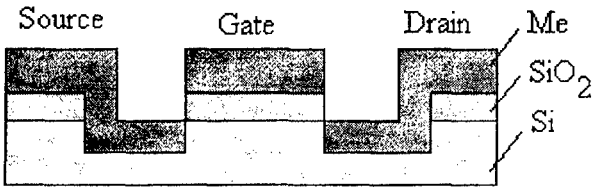

Fig. 2. Setup of a Si/SiO_2 used in a MOS transistor.

The physical structure of surfaces and ultrathin layers has emerged as a critical concern. For layer thickness < 5 nm and surface and interface roughness on the subnanometer scale, some standard methods of surface inspection and thickness control operate partly at their capability limits.

3. Investigation methods

A surface roughness value of 0.1 nm is not far from the noise level of atomic force microscopy [3]. Ellipsometry, a widely appreciated method to determine layer thickness, is not able to quantify thickness of changes of optical constants within thin layers [7, 8]. To avoid confusion, one has to keep in mind that the detection limit, which is frequently quoted to demonstrate the sensitivity of methods like atomic force microscopy, X-ray photoelectron spectroscopy or ellipssometry, refers to the lower limit of qualitative detection of thin layers and roughness under ideal conditions [5, 6]. X-ray scattering from surfaces and thin layers is a valuable tool to determine structure on the nanometer scale quantitatively and nondestructively. Its application to downsized semiconductor features of the near future is based on the weak interaction of subnanometer X-ray waves with matter. The sensitivity of X-ray to lateral and vertical density changes allows one to investigate surface as well as bulk properties of samples. In this way, the intensity and angular distribution of scattered X-ray allows one to calculate surface and interface roughness, densities as well as density profiles, thicknesses of layers and lateral properties such as the surface correlation length and the fractal dimension.

4. Surface parameters

For the Sb_2O_3/SiO_2 sample investigation, it is recommended to use the fractal description of surface. Fig. 3 provides a surface profile that changes its appearance because of changing fractal parameters. In figure, the mentioned parameters are: L is the correlation length, h is the hurst parameter and D is the fractal dimension. The surface roughness, σ, denotes the half-width of the distribution of height fluctuations ($z-z_0$) at the mean height (z_0) of the surface. For the Sb_2O_3/SiO_2 surface, the distribution can be described as a Gaussian. The parameters L and h provide information on the lateral structure of surfaces. As can be seen, the correlation length, l, is a measure for the length scale where σ does not change any more. The Hurst parameter, h, can be understood by comparing the three surface profiles in Fig. 3, where σ and L are kept constant. A decrease of h can be described by an increase of high-frequency, low-amplitude contributions to the surface profile.

The decrease of h does not affect σ, although the surface profile looks more jagged for lower h. For surfaces in general, D calculated to $D = 3-h$. D can be explained as a measure for the capacity of a fractal object to fill the space in which it is embedded.

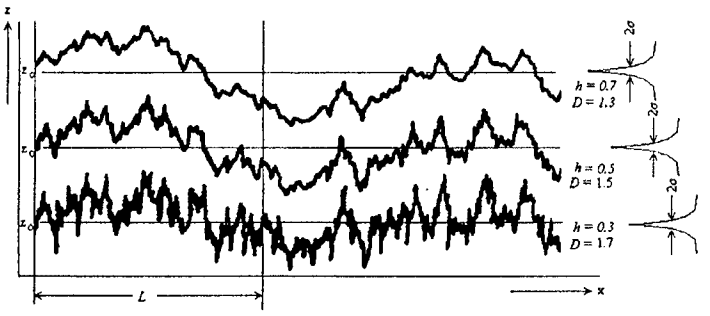

Fig. 3. Fractal parameters of a roughness surface.

5. Instrumentation

The X-ray scattering experiment from the Sb_2O_3/SiO_2 surface was accomplished at the reflectometer displayed in Fig. 4. It consists of an X-ray source; a curved, graded multilayer mirror on the incident beam side; a slit, S_1, in front of the sample mounted onto the Ω circle of the goniometer; and two slits S_2, S_3 preceding the detector on the 2Θ circle of the diffracted beam side.

The X-ray source was a sealed 1.0 kW Cu tube providing an 10 mm×0.05 mm line focus at a 6° take-off angle. The line focus was positioned in the focus of the parabolically curved, grade multilayer mirror acting as beam condenser. Additionally, the mirror plays role of monochromator and separates the $CuK\alpha_1$ and $CuK\alpha_2$ lines for the scattering experiments. For small scattering angles, no further energy resolution was necessary. The resulting wavelength was 0.154 nm. The purpose of S_1 was to cut out a 0.2 mm×10mm part of the incident beam for the scattering experiment. By reducing the beam width from 1 mm to 0.2 mm by S_1, the intensity decreased by a factor of five. This meant a loss for specular X-ray scattering, but it was necessary for the measurement and the interpretation of diffuse X-ray scattering. On the diffracted beam side, S_2 reduced the background scatter. S_2 was set to 0.4 mm×10 mm, and S_3 was set to 0.3 mm×10 mm. With this setup and an instrumental angular resolution of 0.06°, the primary beam intensity received at the scintillation counter detector was 10^6 cps. Eight orders of magnitude proved to be sufficient for the characterization of smooth Sb_2O_3/SiO_2 surfaces.

6. Experiments, results and discussion

Specular X-ray scattering is sensitive normal to the sample surface. It provides vertical structure parameters, i.e., the density and roughness of the Sb_2O_3 surface as well as the density, thickness and roughness of top of SiO_2. The Fig. 4 displays the scattering geometry. By increasing Ω, the scattering signal is recorded by the detector at the angular position $\Theta = \Omega$. The intensity distribution vs. θ, appears as a decreasing curve from $\Theta = 0°$ up to $\Theta = 8°$.The exact shape of the specular signal is determined by the vertical structure parameters. To get information about the lateral structure of the surface, the diffuse scattering has be measured. Hereby, the angle between incoming beam and outgoing beam (2Θ) remains fixed. The angle between incoming beam and sample surface (Ω) is changed from Ω = 0° to Ω = 1.5°. The sharp peaks in the middle of the diffuse scattering curves ($\Omega = \Theta$) belong to the specular scattered intensity. The respective width of 0.03° is determined by the instrument angular resolution of the setup.

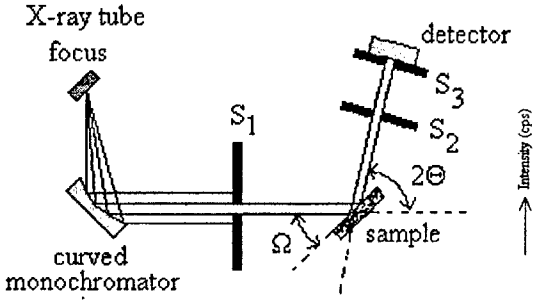

Fig. 4. The setup of the reflectometer.

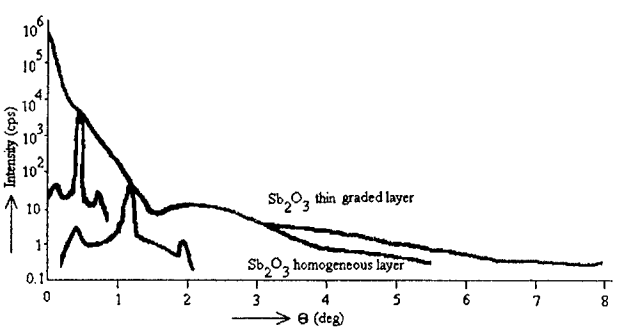

Fig. 5. Specular and diffuse X-ray scattering curves.

The specular scattered signal has some distinct features: From $\Theta = 0°$ to $\Theta = 0{,}2°$ the intensity is nearly constant, with a count rate of 10^6 cps. The negative slope of the signal is affected by parameters

such as the Sb_2O_3 surface roughness, the Sb_2O_3/SiO_2 interface roughness, the density contrast between Sb_2O_3 and SiO_2 and the thickness of the Sb_2O_3 layer. The diffuse scattered signal originates solely in the imperfections of samples. With no surface or interface roughnesses, there would be no diffuse scattering. The small peaks on either side of the diffuse scattering curves in Fig. 5 originate from total external reflection; they are called Yoneda wings. With the exception of the shape specular peaks, the shape of the diffuse scattering curves is determined mainly by the Sb_2O_3 surface roughness, by the lateral correlation length of the surface and by its fractal dimension. The linear grading affects the roughness value of the Sb_2O_3/SiO_2 interface. With 0.02 nm, the interface roughness is below the determination limit of the X-ray scattering method. The thickness value of the Sb_2O_3 layer of 3.0nm demonstrates the sensitivity of the X-ray scattering method to ultrafin structures <10 nm. An intensity of 10^6 cps should be sufficient to quantify even thinner layers. The surface correlation length of 145 nm shows that the X-ray scattering model is wel suited to characterize dielectric structures on the nanometer scale, even laterally. The grading of the 3.0nm Sb_2O_3 layer is the most important result obtained from specular X-ray scattering. This finding is supported by the diffuse X-ray scattering measurements where the scattering signal turns out to be sensitive mainly to the Sb_2O_3 surface shape. The Sb_2O_3/SiO_2 interface looses its scattering contrast because of Sb_2O_3 layer grading, which starts at Sb_2O_3/SiO_2 and ends at the Sb_2O_3 surface. The layer grading reveals that oversimplified assumptions about the homogeneity of thin layers need to be reconsidered. The density values of the graded Sb_2O_3 are 4.88 g/cm^3 at the interface and 4.36 g/cm^3 at the surface. Compared to the oxide's theoretical density of 5.65 g/cm^3, the measured values are very small but quite common for natural oxides. The effect of the thickness and homogeneity on the deposed layer on the specular scattered X-ray curve is presented in the same Fig. 5. Up to $\theta = 3^\circ$, and within an intensity range of seven orders of magnitude, both models yield appropriate fits to the experimental data. Between $\theta = 3^\circ$ and $\theta = 8^\circ$, the models differ.

7. Effects of surface parameters on device performance

As mentioned, real structure parameters affect the proper performance of downsized a sanwich structures. A bite further away from mainstream science, theorist have already shown the dependence of oxide growth on fractal dimension, which has been confirmed by experiment. The precise control of growth processes is crucial for the fabrication of ultrathin layered structures, and therefore, investigations of appropriate models to describe growth processes on atomic scales under nonequilibrium conditions continue. The homogeneity of the oxide density determines the dielectric constant of the oxide. In this way, undesigned density changes of the thin layer can degrade its insulating function.

8. Conclusions

1. In this paper, it is shown that for SiO_2 wafers with an ultrathin Sb_2O_3 layer on top, X-ray scattering is a method for future quality control in the front-end processes of dielectric production.. The method is sensitive to density contrasts and can therefore be applied to all sorts of unpatterned surfaces and layered structures. X-ray scattering is nondestructive and it is applied to structures on the nanometer scale and roughnesses on the subnanometer scale. Because of its sensitivity to density fluctuations, X-ray scattering reveals the inner density structure of ultrathin layers.

2. The results have shown that at least ultrathin Sb_2O_3 layers reveal a graded layer structure. The precise knowledge of the latter is critical for the fabrication of thin layers, and it is crucial for other method of thickness determination that have to rely upon densities and optical constants of layers.

3. X-ray scattering should can be considered as a standard methods for ultrathin layer and surface/interface characterization.

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